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Microstructure and mechanical properties of ARB processed Mg-3%Gd alloy

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Abstract. Mg alloys have various advantages. However, the low formability due to the poor ductility of Mg alloys limits their engineering applications. In this study, an Mg-3%Gd alloy was chosen to explore processing approaches for improving its strength and ductility combination. The alloy was processed by accumulative roll-bonding (ARB) at 400°C to 4 cycles followed by annealing at various temperatures. The microstructures after annealing were characterized by the electron backscatter diffraction technique and the mechanical properties were measured by a tensile test. It was found that the alloy has a good combination of strength and ductility after 2 cycle ARB processing followed by annealing at 290°C for 1h. The strength is 2.3 times higher than that of the fully annealed coarse grained alloy, and the elongation is comparable with that of fully annealed coarse grained counterpart. The good mechanical properties were related to the fine-sized heterogeneous microstructures and weakened texture.

1. Introduction

There has been an increasing demand for magnesium (Mg) alloys as light-weight structural materials in automobile industries during the last decade, mainly due to their various advantages, such as high specific strength, light weight and good dump resistance. However, commercial magnesium alloys usually exhibit poor ductility at room temperatures because of a lack of sufficient number of slip systems. The most effective method to overcome this major drawback of Mg alloy is modifying their microstructures and textures. As is well known, severe plastic deformation (SPD) techniques, such as equal channel angular extrusion, differential speed rolling, large strain hot rolling and accumulative roll-bonding (ARB), have been proved to be effective to weaken the basal texture or to obtain a modified microstructure in Mg alloys. However, ARB is the most prospective SPD method, which could be adopted by industries as a continuous process to produce large sheets with ultrafine grains [1]. The microstructures and texture of the AZ-series Mg alloys processed by ARB method have been well studied in the past decade [2-4]. Recently, hot processing and post-deformation annealing has been proved to be a significantly effective to weaken the textures of Mg alloys containing rare earth (RE) elements, such as Nd, Ce, Gd and Y [5, 6]. In this study, an Mg-3%Gd alloy was selected as an



experimental material and processed by ARB. The mechanical behaviour and microstructural evolution upon annealing of this alloy are studied.

2. Experimental

The material used in this study was an Mg-3%Gd alloy containing 96.86wt.% Mg, 3.0wt.% Gd and 0.14wt.% Al. The cast ingot was extruded at 500°C into ARB starting sheets with a thickness about 1mm, and then annealed at 500°C for 3h. After degreasing and wire-brushing followed by preheating at 400°C for 8 minutes, two stacks of starting sheets were rolled to a 50% thickness reduction. Then, the rolled sheet was cut, stacked and preheated at 400°C for 8 minutes, and then roll-bonded again. Such a procedure was repeated for up to 4 cycles, $\epsilon_{VM} = 3.2$. Rolling was performed in a rolling mill with two 230mm diameter rolls that rotate at 35r/min. The rolls were neither lubricated nor heated and samples were air-cooled after rolling. The subsequent annealing was performed for 1 hour at four different temperatures 190°C, 250°C, 290°C and 400°C.

The microstructures were observed in a Zeiss Auriga dual beam station. The specimens were etched in a solution of 5g picric acid, 10ml H₂O, 70ml ethanol and 10ml acetic acid and local crystallographic orientations were characterized using an Oxford HKL Channel 5 electron backscatter diffraction (EBSD) detector attached to the microscope. For EBSD measurements, the specimen surface was mechanically polished followed by electrochemical polishing in the AC2 solution. Tensile testing was carried out at room temperature under a constant cross-head speed of 0.5mm/minutes. Tensile specimens with 10mm in gauge length and 5mm in width were cut from the ARBed sheets, with the gauge length direction being parallel to the rolling direction (RD). Vickers microhardness (Hv) was measured on the longitudinal cross-section of the samples by imposing a load of 3N dwelling for 10s.

3. Results

3.1. Starting material

The microstructure of the starting sheets before ARB process (after annealing) is shown in Figure 1a. It is seen that the grains are equiaxed with an average grain size of about 40µm. The (0002) pole figure (Figure 1b) of the starting sheets determined by EBSD shows a basal texture, which is typical for Mg alloys.

3.2. Mechanical properties of ARBed and annealed samples

Figure 2 shows the microhardness of the material after ARB processing. It is seen that the microhardness of the sample increases from 43 to 62 during the first pass of the ARB processing; and a very small change is observed during the following passes. Therefore, it is believed that grain refinement has taken place mainly during the first pass, as also shown in [2] that an ultrafine grained microstructure ($d < 1\mu\text{m}$) in AZ91 was obtained during the ARB process under similar rolling condition.

Figure 3a shows the tensile curves of the ARBed sheets. Similar to the tendency of the microhardness curve, the yield strength increases dramatically during the first pass, and then shows little change during the following passes. It is noted that the elongation after the 2nd ARB cycle is slightly better than other cycles.

Figures 3b-d show the tensile curves of the sheets after 1, 2, and 4 ARB cycles and annealed at different temperatures for 1 hour. For comparison, the curve of the starting material is also included in these figures. The low yield strength of the starting sample is attributed to the large grain sizes (Figure 1a). After ARB processing, the yield strength of the material increases greatly and the elongation decreases, which are commonly observed in metals and alloys deformed by ARB. However, the strength increases to even higher values after annealing at 190°C and 250°C. The possible reason could be the segregation of Gd on twin boundaries [7] or grain boundaries [8]. After annealing at 290°C, the

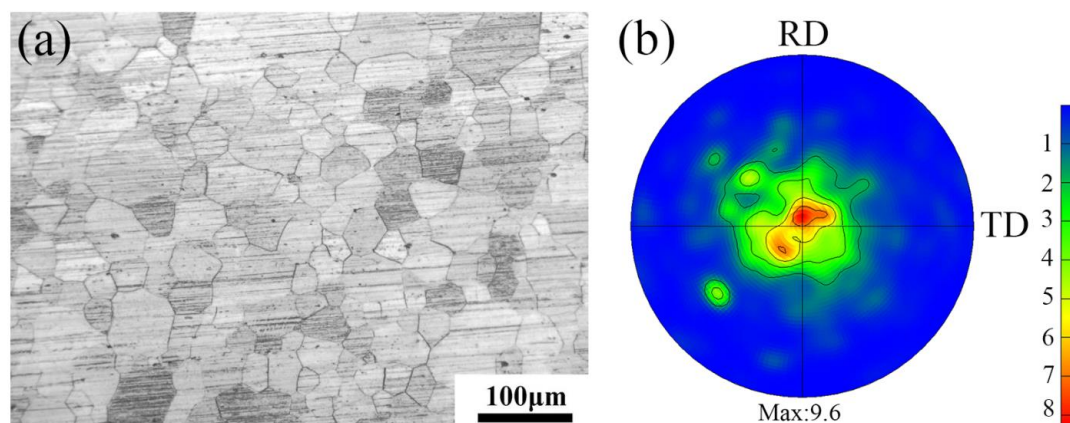


Figure 1. OM micrograph (a) and (0002) pole figure (b) of the starting material for ARB processing.

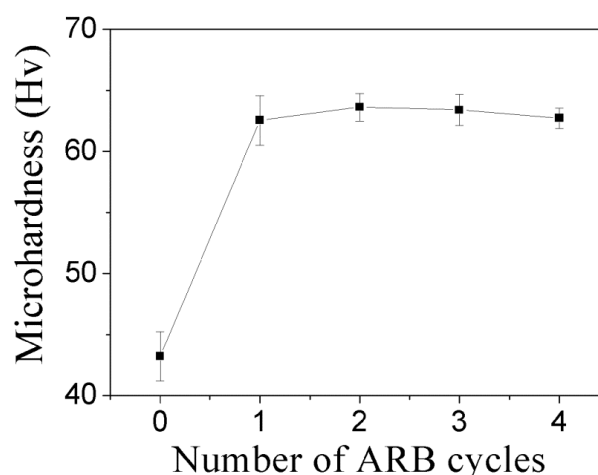


Figure 2. Curve of microhardness of the material after ARB processing.

yield strength of the sample decreases slightly compared with the deformed state. However, the elongation of the sheets increases significantly, close to that of the starting alloy. After annealing at 400°C the yield strength decreases a lot, close to the as-annealed state, and the elongation is almost constant. The mechanical properties of the 2nd and 4th ARBed materials (shown in Figs. 3c and 3d, respectively) are very similar to those to 1st ARBed one. The only difference is that the 2nd ARBed material has a slightly larger elongation after ARB and annealing at 290°C and 400°C.

3.3. Microstructure and texture of annealed samples

It is rather difficult to obtain EBSD patterns with good quality on Mg alloys after deformation to large strains. No EBSD map is available on the ARBed and low temperature annealed samples. Since the mechanical behaviours of the alloy are very similar after ARB processing and subsequent annealing, only the microstructures and texture of the 2nd ARBed sample were characterized.

Figure 4a shows the microstructure from one surface to the center of the sample after annealing at 290°C. A typical heterogeneous microstructures with a well recovered matrix mixed with fine-sized recrystallized grains can be observed in the sample. The size of the large grains is about 10 μm whereas the small grains have grain sizes of about 1 μm. Figure 4b shows that the sample is fully recrystallized

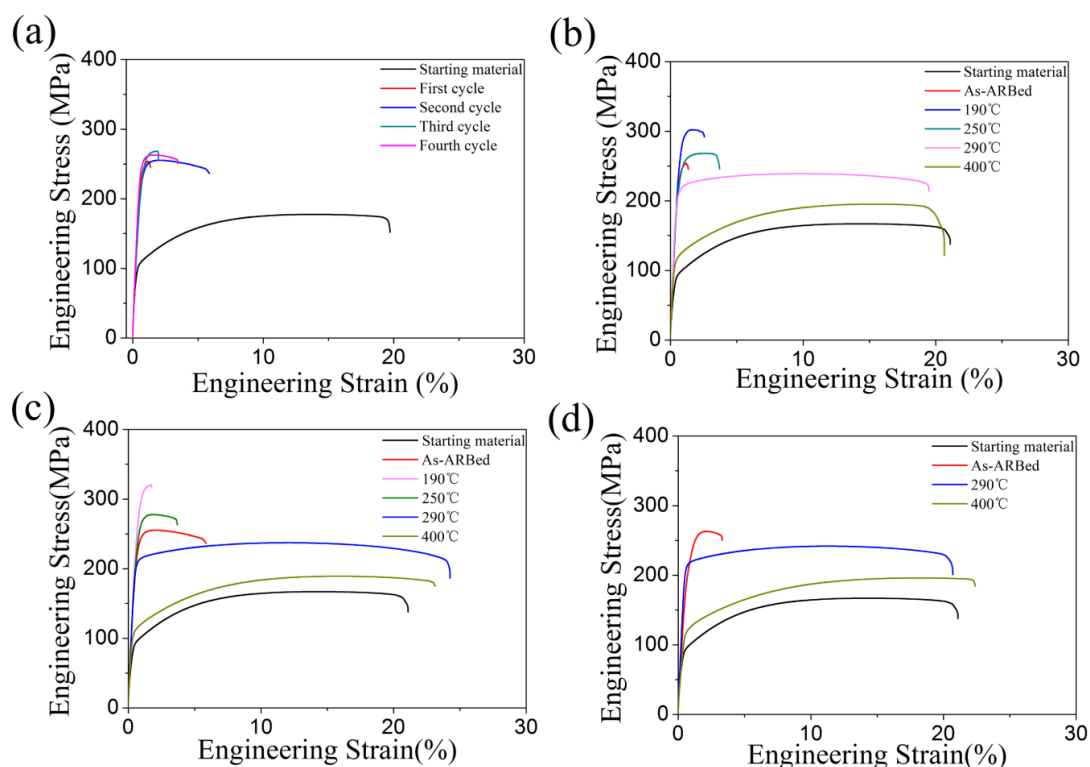


Figure 3. Tensile curves of (a) the ARBed sheets and 1st (b), 2nd (c) and 4th (d) ARBed sheets after annealing at different temperatures.

after annealing at 400°C, exhibiting equiaxed grains with an average size of $\sim 15\mu\text{m}$. As shown in Figure 4, the interface introduced by the first cycle can still be observed. Although the size of grains at the interface is smaller than those in other areas, there is no obvious gradient of grain size across the thickness of the samples annealed at 290°C and 400°C. Figure 5a shows that the texture of the sample annealed at 290°C is the basal texture. Compared with the texture of the starting sheet (Figure 1b), the c-axis of the maxima rotates 15° away from the ND towards the negative RD. As observed in the Figure 5b, a rather weak basal texture is formed in the sample after annealing at 400°C and the texture is much more spread.

4. Discussion

4.1. ARB processing effect on mechanical properties

The present results demonstrate that the strength and hardness were enhanced due to the large strain imposed on the sheet mainly during the first cycle (Figure 2 and Figure 3a). It is known that grain growth in Mg alloys is rapid when the ARB temperature is higher than $0.5T_m$ (the melting point) or 461K or 188°C, thus the repeated heating at 400°C between rolling cycles is above the recrystallization temperature of magnesium can result in static recrystallization and partially eliminate the accumulated strain. It has found that the interval reheating of pure Mg can lead to full recrystallization and a large grain size [9, 11]. In this study, the intermediate annealing leads to saturation of hardening already after one cycle.

ARB introduces a large shear strain in the sheet thus the microstructure is greatly refined. At the same time the texture is also weakened during the subsequent annealing at 290°C and 400°C due to recovery and static recrystallization. After annealing at 290°C, the microstructure is greatly refined.

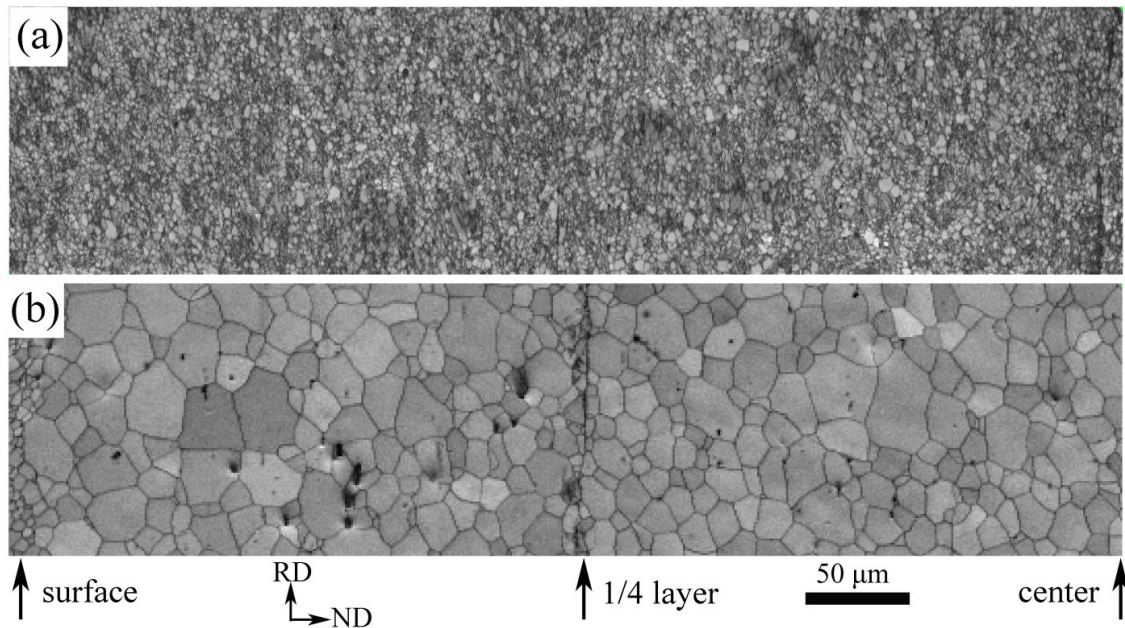


Figure 4. Microstructures of the materials annealed at 290°C (a) and 400°C (b).

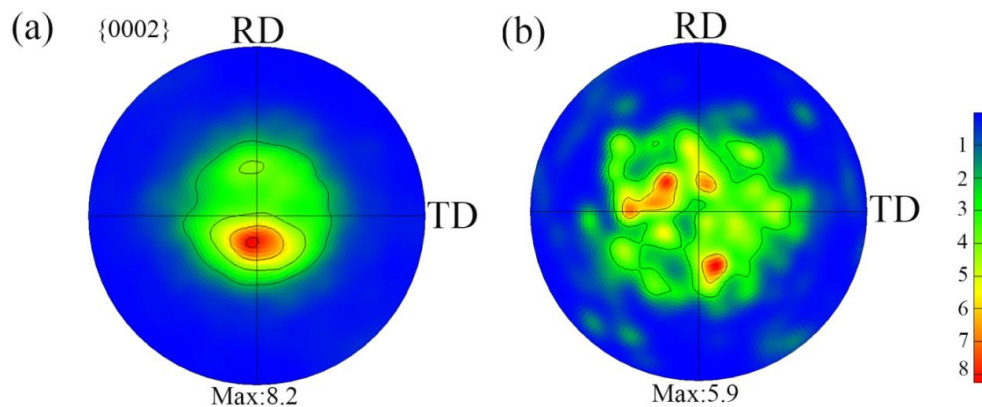


Figure 5. (0002) pole figure of the samples annealed at 290°C (a) and 400°C (b).

The best combination of elongation and strength is achieved, with the yield strength being 210 MPa and total elongation 24%. Compared to the mechanical properties of the starting Mg-3%Gd alloy, the yield strength is 2.3 times larger while the elongation is about the same (even slightly larger). In another study, the yield strength and elongation of an Mg-3(wt.) %Gd with an average grain size of 76μm after solute treatment at 535°C for 1.5 hours are 71MPa and 13%, respectively [10]. For an Mg-Gd-Zn alloy with similar compositions (3%Gd, 1%Zn), the highest yield strength of 143MPa and elongation of 30% were obtained after warm rolling at 430°C to 85% reduction and annealing at 350°C [11]. Therefore, the combination of strength and elongation of the present Mg-3%Gd alloy is greatly improved by ARB processing and subsequent annealing. Compared with other ARB processed commercial Mg alloys, such as AZ31, the present Mg-Gd alloy has much better mechanical properties. For example, optimized yield strength and total elongation are 160 MPa and 20% in an AZ31 alloy after 2 cycle ARB processing at 450°C and annealing at 350°C [1].

4.2. Grain size and texture effect on mechanical properties

The elongation of the ARB processed Mg-3%Gd alloy does not increase through annealing at temperatures below 290°C. Greatly improved elongation is obtained after annealing at 290°C. This is attributed to the occurrence of static recrystallization and the presence of new fine recrystallized grains in the microstructure. Extensive static recrystallization takes place in the microstructure at 290°C. Thus, the increased volume of fine sized recrystallized grains from the previous ARB process will improve the elongation of the alloy, which is better than the elongation of the sheet annealed at 400°C with a grain size of 15µm. It is interesting to note that the sheet annealed at 290°C has a strong basal texture while the 400°C annealed sheet has a more spread and weak basal texture. It was suggested that the modified texture would introduce an excellent room temperature ductility in Mg-RE alloys sheets and that the grain size has a negligible effect on room-temperature ductility compared with texture [12]. However the present study shows the beneficial effect of grain size is worth a further investigation.

Figure 6 shows the EBSD maps taken from the necking zones close to the fracture. Figure 7 shows the misorientation angle distribution of the sample before and after the tensile test. Both figures show that twinning is activated during the tensile test of the sample annealed at 400°C. It is well known that grain size has a significant influence on twinning activity [13]. The effect of grain size on ductility in AZ31 alloy processed by equal channel angular extrusion and rolling was studied, and it was found that a transition from slip-dominated to twinning-dominated flow occurs with increasing grain size [14]. They have shown that the twinning activity of the rolled AZ31 sheets obviously increases when the grain size increases from 4.2 µm to 17 µm. When the grain size is larger than 20 µm, the deformation mode of the AZ31 sheet even turns to be twinning-dominated flow. The 400°C annealed sample has a coarse grain size. In this sense, the twin activity of the 400°C annealed sample fits with the results in the literature. Although the 400°C annealed sample with a more random texture favours slip during deformation, the deformation mode is twinning-dominated flow after deformation due to the large grain size.

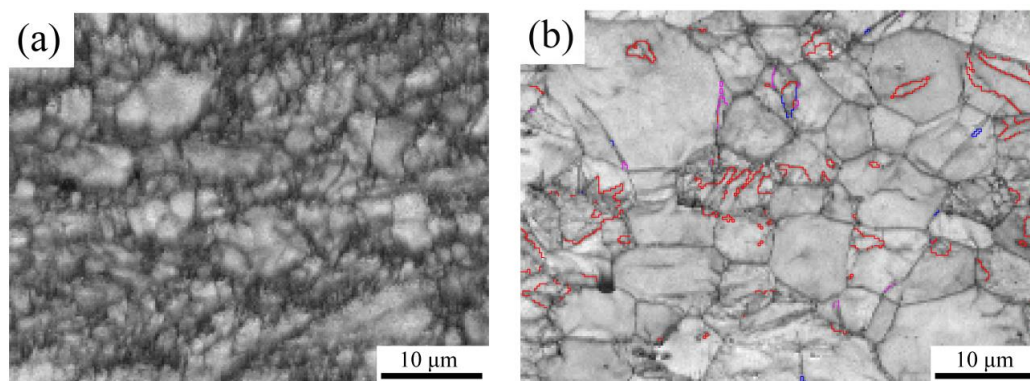


Figure 6. The EBSD maps of (a) the 290°C annealed sample and (b) the 400°C annealed sample taken from the necking zone close to the fracture. The red line, blue line and purple line indicate extension twinning, double twinning and contraction twinning, respectively.

It is seen that the frequency of low angle grain boundaries in the 290°C annealed sample is higher than that of the fully annealed sample, except for the first data point, which is believed to be related to the rather low angular resolution limitation of the EBSD technique (Figure 7b). In comparison with the 400°C annealed sample, the twinning activity of the sample annealed at 290°C is negligible. This

suggests that slip of dislocations is the deformation mechanism. Although the basal texture is rather strong in the 290°C annealed sample, the fine grain size may facilitate slip activity.

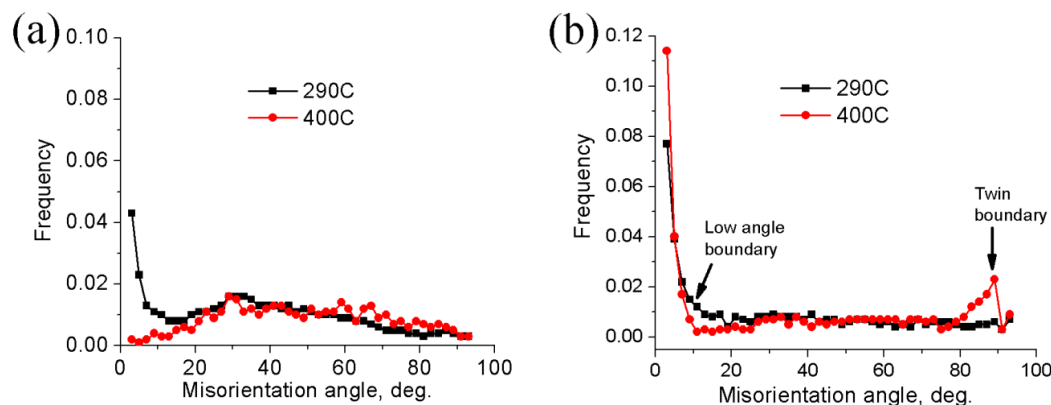


Figure 7. The misorientation angle distribution of the sample before (a) and after (b) the tensile test.

5. Summary

A magnesium alloy containing 3wt.% Gd and 0.14wt.% Al alloy was processed by ARB at 400°C up to 4 cycles. The microstructure, texture and mechanical properties of the ARBed Mg-3%Gd sheets were studied and the main findings are the following:

- Mechanical properties (yield strength and tensile elongation) were improved dramatically through ARB and post-process annealing at 290°C.
- The sample annealed at 290°C develops a heterogeneous microstructure, a mixture with fine (1µm) and coarse grains (10µm). It has a 2.3 times higher yield strength than that of the fully annealed coarse grained alloy; and the elongation is almost the same, about 24%.
- It is suggested that there is a transition from twinning-dominated deformation to slip-dominated deformation when the grain size changes from 15 µm to 2.6 µm in the present Mg-3%Gd alloy.

Acknowledgments

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